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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
Contract NAS 3-13004
James Sovey, Project Manager

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FINAL REPORT

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ABSTRACT

The application of glass coatings to molybdenum ion accelerator grids by the electrophoretic deposition technique was demonstrated. The process involves control of the characteristics of the glass-water suspension and deposition parameters of voltage, current, deposition time, and cathode-anode spacing. The technique permits deposition of the required thickness in a single application. Seven- and 30-centimeter grids were coated and heat treated to yield nearly bubble-free glass-coated composite grids.

SUMMARY

Glass-coated molybdenum ion accelerator grids were prepared by depositing the glass coating electrophoretically in a single deposition process and fusing the glass by a previously developed atmosphere-heating schedule to give a nearly bubble-free coating.

Deposition of the coating was accomplished by immersing the grid as the anode and a bronze cathode of like configuration into a glass powder-water suspension. Application of a direct-current potential between the two electrodes facing each other and several inches apart caused glass particles to migrate to and deposit on the molybdenum anode. A second electrochemical reaction involved the formation of molybdenum ions at the anode and liberation of hydrogen gas at the cathode. Deposition conditions were optimum when the total current comprised the proper balance between glass-particle current carriers and ion current carriers. Under optimum conditions the glass coating formed on the molybdenum and was bonded to it apparently through a flocculating action of molybdenum ions. The important deposition parameters included voltage, current, deposition time, specific gravity of the glass-water suspension, and electrode spacing.

It was found to be advantageous to coat the hole walls and both faces of the grid in the interest of building up the maximum coating on hole wall surfaces. The coating deposited on the back face was removed before fusing the coating to the grid. Grids 7 and 30 cm in diameter were coated to give nearly bubble-free glass coatings, 0.056 cm thick, after the fusing treatment.

INTRODUCTION

A main component of an electron-bombardment ion thruster is the accelerator grid system. One such system comprises a screen grid and an accelerator grid. The two must be precisely aligned relative to one another and remain aligned during thruster operation. An alternate system consisting of an electrically insulating glass coating on a metal grid substrate has several advantages over the two-grid system, one being the elimination of the need for alignment. Details of the grid system, as well as a process for fabricating a glass-coated grid, have been described by Banks.[1, 2]

In the work referred to, a water suspension of glass powder was applied to one face of a molybdenum grid by the conventional wet-spraying process. The glass coating was fused to the grid by a special atmosphere heat treatment that resulted in a coating virtually free of gas bubbles. This in turn resulted in a glass coating of enhanced electrical resistivity. Evaluation of such glass-coated grids demonstrated the concept to be feasible and advantageous.

The present work was concerned primarily with an alternate technique for applying the glass to the grid. The method studied for coating the grid was that of electrophoretic deposition or, more simply, electrodeposition. The wet-spraying technique of applying the coating, while workable, required a number of applications with a firing operation after each. This was necessary because the relatively thick glass coating

required could not be applied in a single application. The electrodeposition technique was studied with the aim of applying the total coating thickness in a single operation.

Using the electrodeposition method, the grid to be coated is arranged to face a second metal element of similar configuration at a short distance from it. The two metal pieces are connected to a direct-current source so that the grid is the anode and the other piece the cathode. The assembly is immersed in a water suspension of glass powder. Coating takes place when a potential is applied across the metal electrodes. The glass particles become charged and migrate in the electrical field to deposit on the grid anode.

The purpose of the work described here was, ultimately, to provide glass-coated grids 30 cm in diameter for evaluation in a thruster. The procedure was one of determining the processing parameters for coating, first a simple 5.1-cm square sheet, second a grid 7 cm in diameter, and finally the 30 cm-diameter grids.

The significance of the present work is concerned only partially with its successful conclusion. The electrodeposition process for applying coatings of glass or other like materials is not widely appreciated. Of broad significance is the demonstration of a technique for applying coatings where unusual circumstances render more conventional coating processes less attractive or not even practical.

BACKGROUND

The basic process of depositing a coating on a metal substrate by causing charged particles to migrate to the metal electrode under the influence of an electric field is not new, though it has not been widely publicized. Hughes[3] has described the application of porcelain enamels to steel using the electrodeposition process. (Reference is made to this publication for general background on the process.)

Though the process appears simple on the surface, it is in fact complex. The parameters involved in depositing a satisfactory coating of a specific thickness include:

(1) Applied d-c voltage

(2) Current

(3) Deposition time

(4) Electrode configuration and spacing

(5) Specific gravity of glass-water suspension

(6) Temperature of glass-water suspension

(7) Type and amount of current carriers.

All but the last-listed parameter are readily measurable and controllable. Discussion of the current carriers and their effects is reserved for a later section. Suffice it to say here that the charged glass particles are not the only carriers; various ions in the water also participate, to both complicate the process and enhance it.

There are several significant features of the electrodeposition process that distinguish it from more conventional coating processes such as wet spraying or dipping. First, the glass-particle coating is more dense during and immediately after deposition when applied by electrodeposition than when applied by wet spraying. That is, the coating, per se, has a higher glass-to-water ratio in the former case, as only the glass particles are attracted to the metal substrate. Virtually the only water in the deposited coating is that present interstitially in the glass-powder layer. Coatings applied by wet spraying or dipping will contain water up to the same content present in the starting suspension. This would include not only interstitial water but water films surrounding solid particles. This feature is not so significant when coating a simple sheet, but can be highly significant in the case of a perforated sheet where the holes are small and must not be filled in, as in the case of the grids.

A second advantage to the electrodeposition process is its ability to provide coating on thin metal edges and on the corners of right-angle intersections. Again, this feature is attributed to the characteristic of the process whereby the glass particles are attracted to the metal electrically rather than covering the metal by virtue of viscous flow and surface tension characteristics of a water-glass suspension, as in the case of spraying or dipping.

A third feature of the electrodeposited coating, which is significant in the present application, is the bonding of the deposited coating to the metal which is developed under appropriate depositing conditions. No additive can be made to the glass-water suspension to promote coating strength and coating-to-metal bonding, because the conventional additives such as clay or organic compounds would be a source of bubbles in the fused-glass coating. They would evolve gases at the high fusing temperatures.

Another potential feature of the electrodeposition process is the uniformity of coating thickness attainable. The configuration and location of the cathode largely determines the uniformity. In wet spraying, the skill of the sprayer governs uniformity, and in the dipping process, the draining characteristic of the excess suspension is the governing factor.

COATING STUDIES

The objective in the studies described in the following sections was to process molybdenum substrates to coat them with a bubble-free fused coating of glass 0.056 cm thick. Because of its thermal-expansion match with molybdenum and its relatively high electrical resistivity, Corning glass No. 7052, ground to pass a 100-mesh sieve, was used.

Phase I. Coating Molybdenum Sheet

The coating processes included:

(1) Cleaning the metal surface

(2) Electrodepositing the glass coating

(3) Oxidizing the metal surface

(4) Atmosphere firing to fuse the coating.

The metal surface was cleaned by scouring it with a suspension of 200-mesh quartz in an alkaline detergent. The surface was considered clean when a water film completely covered the metal surface and did not draw up to form droplets. A final treatment was immersing the specimen in distilled water.

The glass coating was deposited by immersing the molybdenum specimen (anode) and a stainless-steel screen (cathode) of the same dimensions as the anode (5.1 by 5.1 cm) into a water suspension of the No. 7052 Corning glass powder. The two electrodes (specimen and screen) faced each other and were spaced 5.1 cm apart. Leads from a d-c rectifier were clamped to the electrodes by alligator clips. The glass-water suspension had a specific gravity of 1.3 attained by dispersing 70 parts by weight of glass in 100 parts water. Suspension was maintained by mildly agitating the glass-water slurry using a laboratory stirrer. After immersing the electrodes in the suspension, voltages in the range of 50 to 150 volts were applied for periods of about 5 to 20 seconds after which the glass-coated specimens were removed from the suspension and allowed to dry.

The dried, coated specimens were then placed in a furnace (air atmosphere) at 537°C for 10 minutes. This treatment developed a purplish oxide film on the metal surface. The fact that the oxide film developed on the metal surface covered by the glass coating was noted by removing the glass after one such heat treatment. This procedure differs from the one described by Banks[1] in that he provided the oxide film before applying the glass coating. This sequence was reversed because in one electrodeposition trial it was observed that the oxide film that was developed before the coating process was removed in the electrodeposition process.

The final operation of atmosphere firing the coated specimens followed the procedures described by Banks.[1] A minor difference involved the use of two furnaces instead of one. This was done to minimize the time between the 30-minute holding period at 610°C and the final heat treatment at 955°C. During the operation the specimen(s) were contained in a nominally 12.7-cm cube stainless-steel box with double lids as used by Banks.

The heating atmosphere schedule used was:

(1) Room-temperature purging in helium atmosphere for 30 minutes.

(2) Holding for 30 minutes in furnace at 610°C – helium environment.

(3) Removing to a furnace at 955°C and holding for 35 minutes – helium environment. Of this 35-minute period, 20 minutes were required to reach 955°C after which the specimen was held 15 more minutes.

(4) Change from helium to argon environment and holding at 955°C for 15 minutes.

(5) Specimen removed from furnace and allowed to cool to 260°C in argon environment.

Temperatures were measured using a thermocouple inserted into a well in the stainless-steel box. Atmosphere gases were preheated by coiling the stainless-steel tubing leading them into the box.

A number of preliminary coating trials indicated that two extreme conditions could prevail which resulted in coatings of poor quality, and that the condition for processing a good coating consisted of a compromise between these extremes.

One of the extreme conditions resulted in a coating which was completely white in color after the deposited glass-powder coating was dried and which adhered poorly to the molybdenum substrate which was virtually unchanged in appearance. In the second extreme case, little or no glass coating was deposited, the molybdenum substrate developed a blackened surface, and a pronounced evolution of hydrogen gas occurred at the cathode. The preferred compromise conditions resulted in a coating characterized by a faint bluish color in the otherwise white glass coating and by good bonding of the coating to the slightly darkened molybdenum substrate.

The various results are attributed to the amount and type of current carriers in the water-glass suspension. Because the conditions for depositing a good coating could be defined by electrical and other deposition parameters, the characteristics of the current carriers were not determined quantitatively. However, the current carriers would be expected to comprise charged glass particles, ions resulting from a slight solubility of the glass in water, molybdenum ions derived from the substrate through the electrochemical process, and hydrogen, oxygen, and hydroxyl ions.

The first extreme case described results when the suspension is free, or virtually free, of ion conductors. Under the influence of the electric field, the glass particles move to the molybdenum anode but do not adhere well to it. In the second extreme case, ions are the major current carriers and the glass particles the minor. In this case, hydrogen ions in the water acquire electrons and evolve as hydrogen gas at the cathode, while the molybdenum metal anode gives up electrons to produce molybdenum ions. Some of these in turn combine with oxygen ions to produce the black molybdenum-oxide coating on the molybdenum metal; others apparently remain in solution in an oxidation state to give a blue solution which diffuses into the glass coating.

The various electrodeposition behaviors were reflected in voltage-current parameters. Starting with a fresh glass-distilled water suspension, the current during deposition at a given voltage was relatively low. This situation describes the first extreme case. With each succeeding deposition trial in which the voltage was maintained constant, an increase in current resulted. Ultimately, with ion buildup from previous trials, the current reached a relatively high value. The main current carriers were ions rather than glass particles, and poor coating resulted.

Optimum results were obtained when the current was moderately low. Under this condition, the glass coating deposited readily and adhered well. The good adherence is attributed to the reaction wherein molybdenum ions, liberated at the substrate, apparently flocculate and bond the glass particles to each other and to the metal surface.

The optimum conditions for coating the 5.1 by 5.1-cm sheet occurred at an applied potential of 100 volts and a current of 400 milliamperes. Under these conditions the relation between deposition time and thickness of the fused-glass coating was:

Deposition Time, sec	Fused-Glass Thickness, cm
8	0.030
9	0.038
10	0.046
11	0.058
11.5	0.071
12	0.089

Figure 1 shows a coated specimen processed under these optimum conditions and fused as described previously. Figure 2 is a view of the same specimen at higher magnification for the purpose of showing the bubble-free character of the glass.

The results of the trials in Phase 1 indicated an optimum set of conditions for successful coating. Of major importance was control of the electrical characteristics of the glass-water suspension. A freshly prepared suspension tended to result in a thick coating of poor adherence. In such a suspension, an applied potential of 100 volts produced a current of less than 200 milliamperes. With ion buildup from previous trials, the coating characteristics improved, reached an optimum, then deteriorated. As currents exceeded around 800 milliamperes, hydrogen evolution was excessive, and the coating dried to a strong blue color that was attributed to excessive molybdenum ion formation. A deteriorated suspension could be corrected by decanting the excessively ionized water and replacing it with distilled water. The optimum suspension was characterized by the 100-volt, 400-milliampere voltage-current parameters.

Phase II. Coating 7-Cm-Diameter Grids

This phase differed from Phase I primarily in the difference in configuration of the molybdenum substrate. The grid to be coated was a 7-cm diameter disc, 0.038 cm thick, with 0.190-cm-diameter holes, spaced 0.254 cm center-to-center. One surface and the hole walls of the grid were to be coated; thickness of the surface coating was to be 0.056 cm. Two types of grids were coated, one having the punched holes and the other having drilled holes. They differed in that the intersection of the hole wall and surface of the former was rounded, whereas the latter had sharp edges. Except where noted, the cleaning, coating, and atmosphere-heating processes of Phase II were the same as those described in the Phase I section.

In initial coating trials, the grid was mounted in a stainless-steel electrode ring as shown in Figure 3. Copper wires tied the grid to the ring. The grid anode was positioned 6.4 cm from a stainless-steel screen cathode. Electrical leads to the two electrodes were through alligator clips.

In the first trials, the stainless-steel anode ring had a plastic disc fastened to its back, since only one side of the grid was to be coated. This disc was attached by three screws as shown in the ring of Figure 3. Subsequently, it was recognized that it would be advantageous to allow the grid to be coated on both sides, and the plastic backing disc was discarded. It was reasoned that simultaneous deposition on front and back faces and hole walls would permit maximum deposition on hole wall surfaces. A coating thus deposited is shown schematically, in section, in Figure 4 (a). After the deposited coating dries and before it is fused to the grid, the coating on the back face

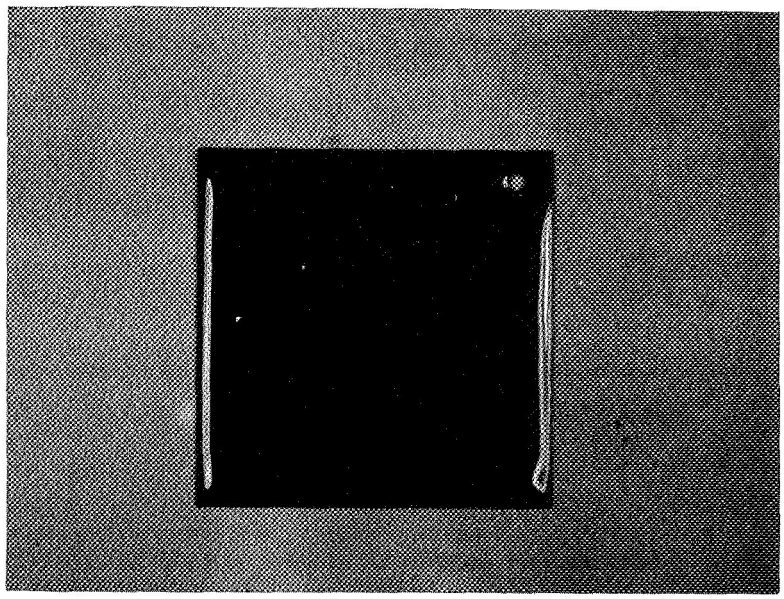


FIGURE 1. VIEW OF SPECIMEN WITH A 0.044 CM COATING SHOWING SURFACE

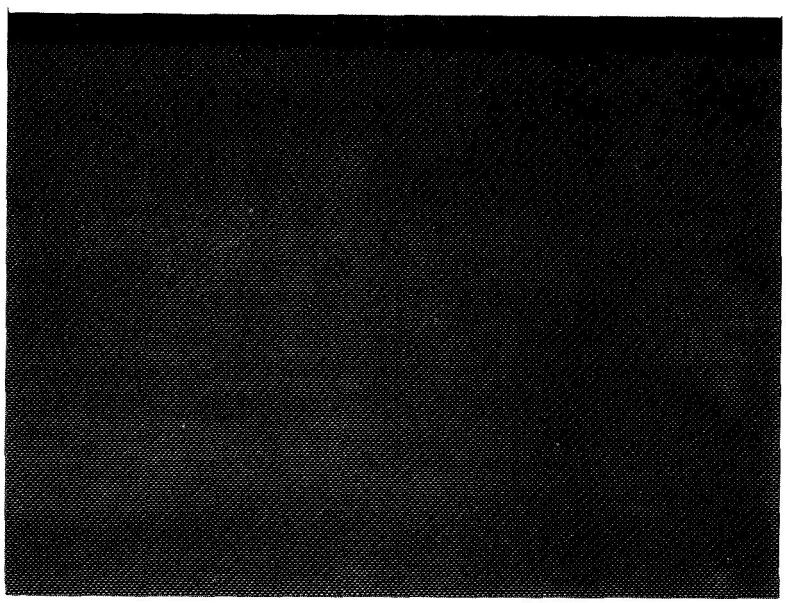


FIGURE 2. VIEW OF SPECIMEN WITH A 0.044 CM COATING AT 10X MAGNIFICATION

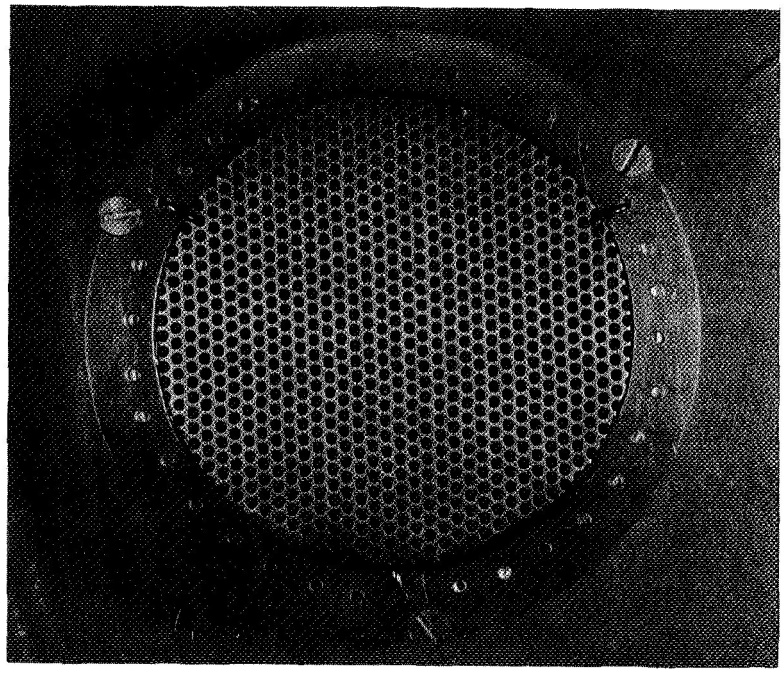


FIGURE 3. ELECTRODE ASSEMBLY OF GRID AS THE ANODE FOR ELECTRODEPOSITION

can be scraped off as shown in Figure 4 (b). During fusing, the surface tension forces of the viscous glass would tend to reshape the coating to produce the desired final configuration shown in Figure 4 (c).

The coating process in this phase was modified slightly from that used in coating the 5.1-cm-square sheets. The sheets were immersed in the glass-water suspension with their plane in the vertical position. The grids were immersed in the horizontal position and positioned above the stainless-steel screen cathode. With their plane in the horizontal position, some glass particles settled, via gravity, to deposit on the top surface of the grid. To remove these extraneous particles, the grid-cathode assembly was immersed in a clear water bath immediately after the electrodeposition treatment. During this rinsing process, an applied voltage of one-half that used during electrodeposition was maintained.

Grids in which the holes were formed by punching were processed under the following optimum conditions:

Specific gravity of glass-water suspension	1.10
Applied voltage	100 v
Current	800 ma
Deposition time	60 sec
Thickness of unfused coating	0.081 cm
Thickness of fused coating	0.056 cm

Figure 5 shows views of a coated grid after electrodeposition but before the coating is fused. View (a) is of the coated front face and view (b) shows the back face after the deposited coating on this side had been scraped from the grid.

Figure 6 shows the same grid after the fusing process.

Grids in which the holes were formed by drilling behaved similarly with the exception that coating thickness of but 0.064 cm was required to produce a grid with a fused coating thickness of 0.056 cm.

Phase III. Coating 30-Cm-Diameter Grids

The facilities used for coating the grids are shown in Figures 7, 8, and 9. Figure 7 shows the arrangement for positioning the anode (molybdenum) and cathode (bronze screen) in preparation for immersing it into the glass-water suspension. Figure 8 shows two plastic 32-gallon trash containers used in the electrodeposition studies. One contained the glass-water suspension. The other contained water only. After removing the anode-cathode positioner from the glass-water depositing container, it was immersed in the water to remove extraneous glass particles from the grid which were loosely held by virtue of their settling on it via gravity rather than electrochemical processes. Figure 9 shows the stainless-steel box, containing coated grids, in position for entering the furnace used for firing the glass coating.

Figure 10 shows one of the coated grids prior to firing. The three uncoated areas on the ring to which the grid was fastened were the places where the ring was in contact with the anode-cathode positioner during the coating process. Figure 11 shows one of the glass-coated grids after firing.

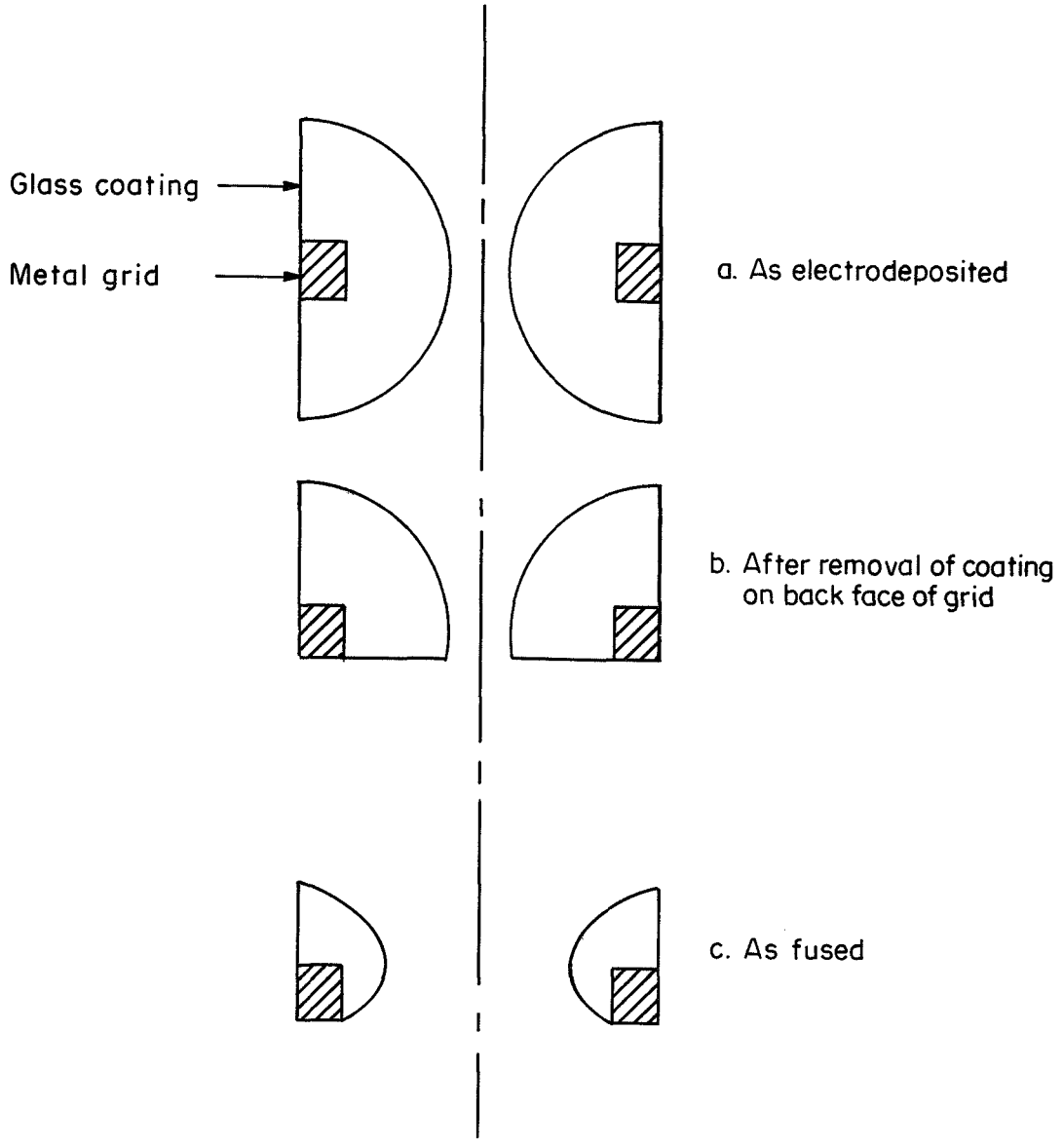
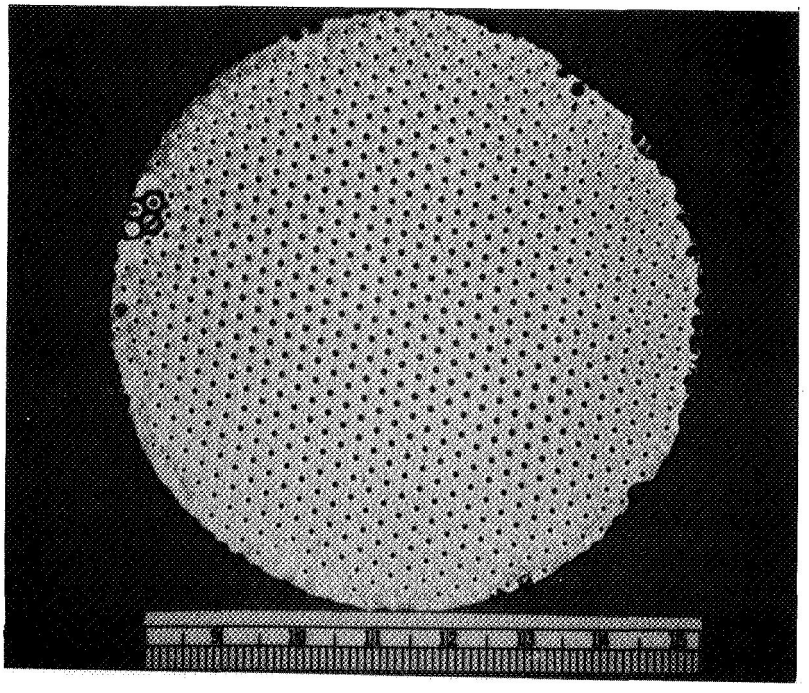
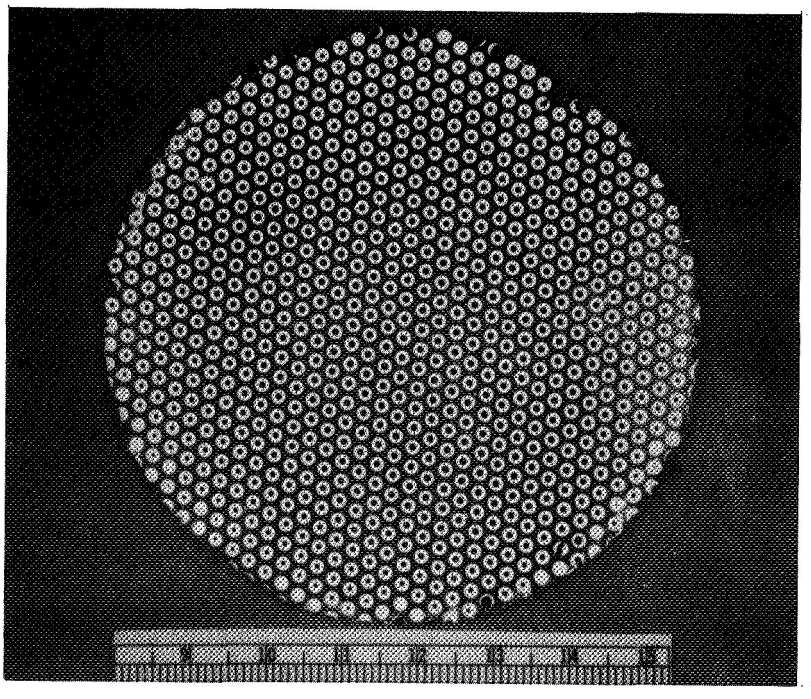


FIGURE 4. SECTION THROUGH GRID HOLE SHOWING GLASS COATING ON GRID

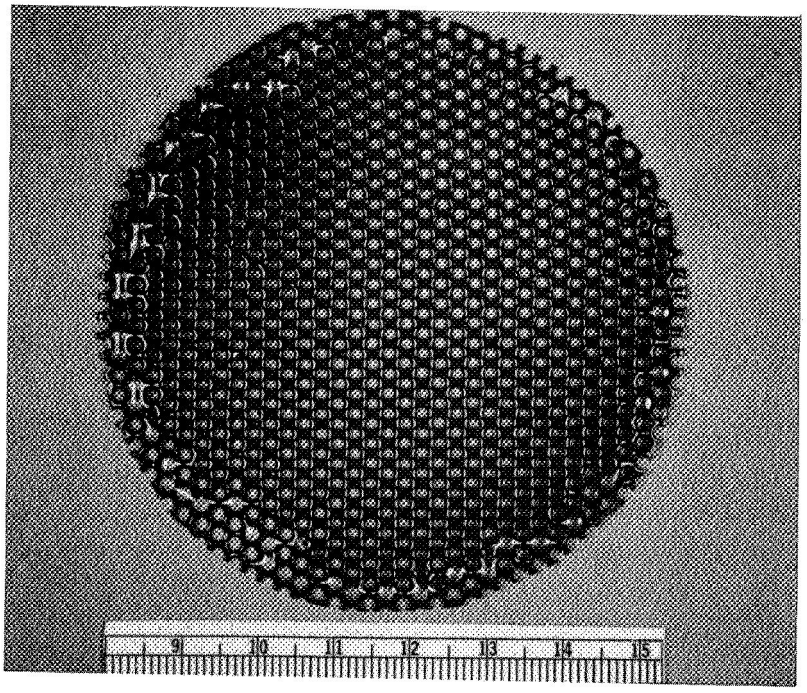


a. Coated Side

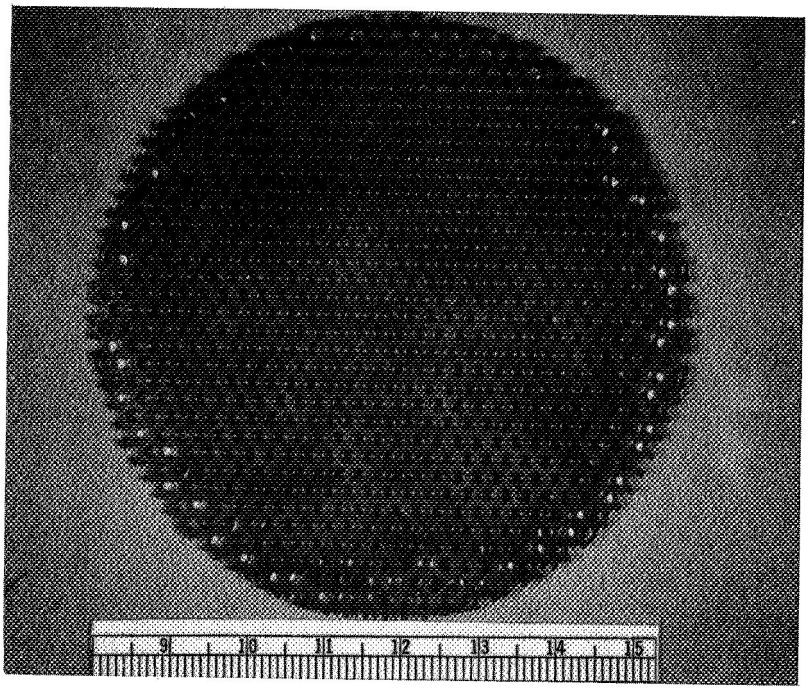


b. Uncoated Side

FIGURE 5. ELECTRODEPOSITED BUT UNFUSED GLASS COATING ON 7-CM GRID



a. Coated Side



b. Uncoated Side

FIGURE 6. FIRED GLASS-COATED 7-CM GRID

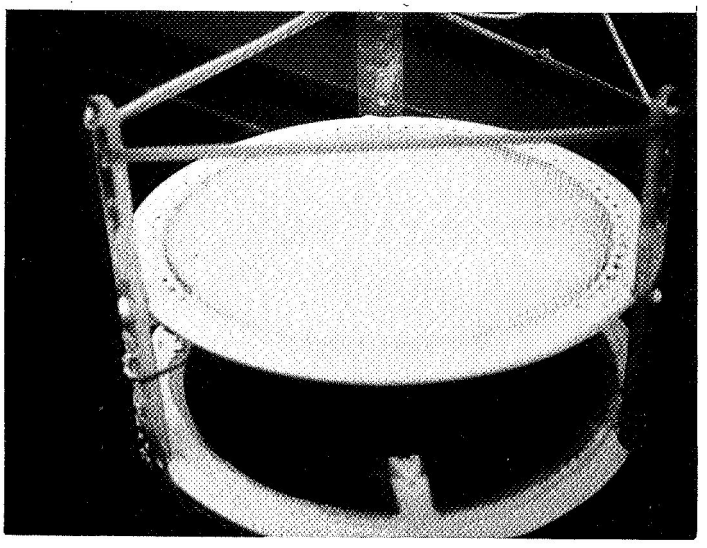


FIGURE 7. ANODE-CATHODE POSITIONER

The coated molybdenum anode is above the bronze cathode.

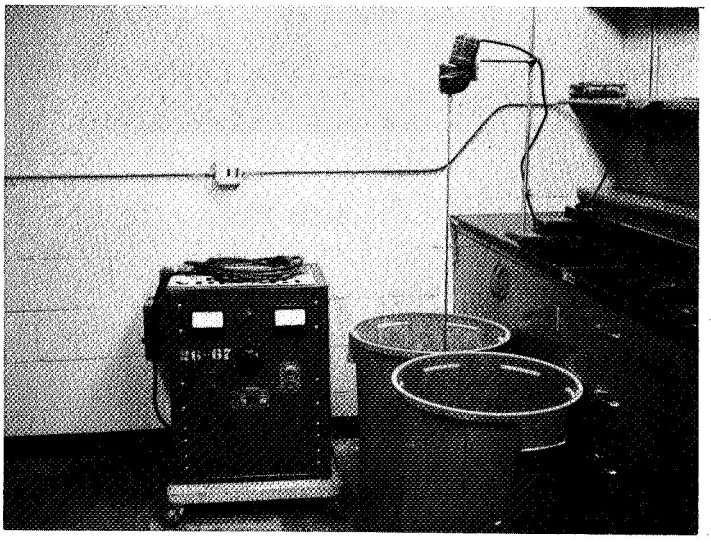


FIGURE 8. ELECTRODEPOSITION AND RINSING CONTAINERS

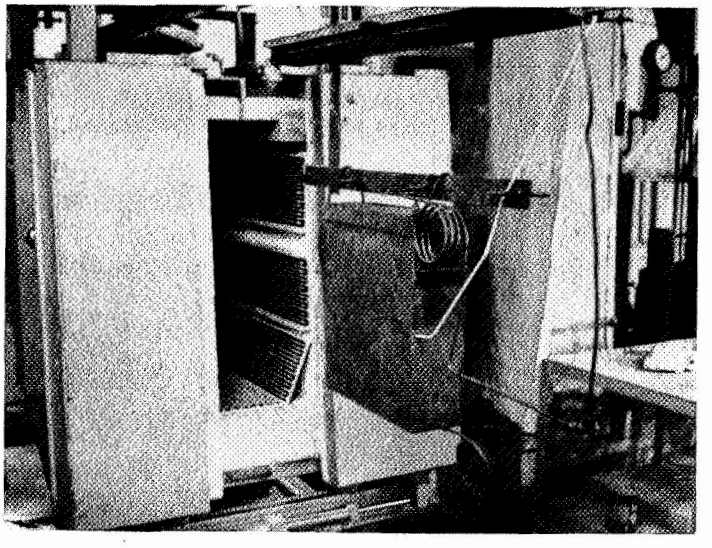


FIGURE 9. STAINLESS-STEEL BOX AND FURNACE FOR FIRING COATED GRIDS

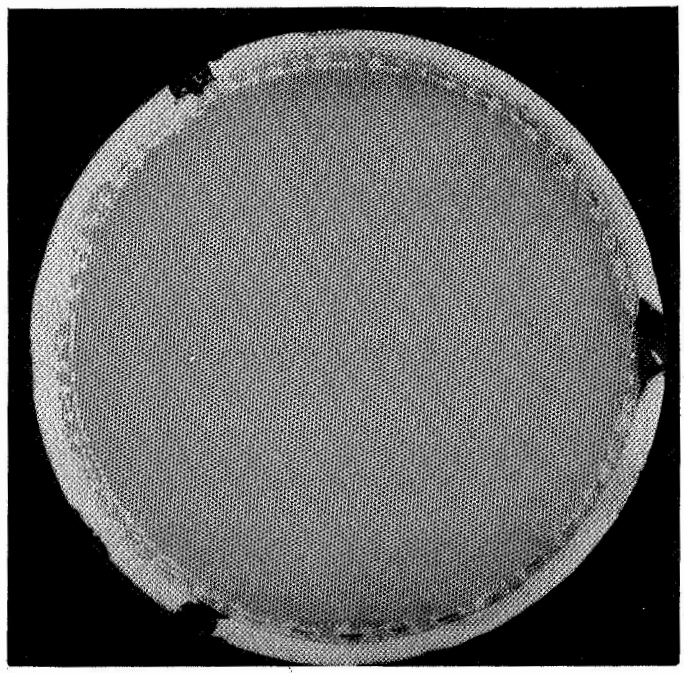


FIGURE 10. COATED BUT UNFIRED GRID

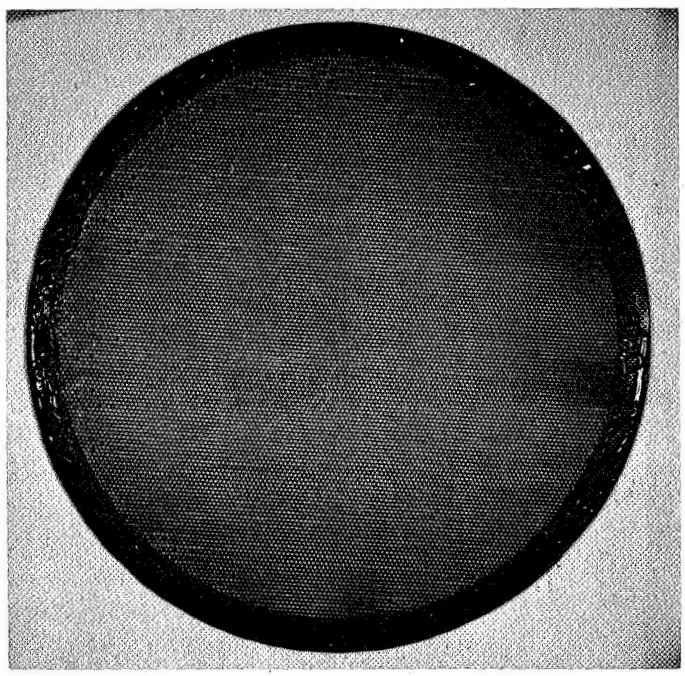


FIGURE 11. COATED GRID AFTER FIRING

Several difficulties were encountered in coating the large grids which were not encountered in coating the 7-cm grids. Because of the way in which the grid was fastened to the mounting ring, air pockets formed under the grid when it was immersed in the glass-water suspension. The ring prevented their escape at the periphery of the assembly when it was immersed with the ring on the bottom. This problem was corrected by immersing the assembly with the ring on the top. This arrangement created the potential for hydrogen bubbles to form on the grid surface to be coated (through their evolution at the cathode immediately beneath the grid) and thus interfere with the coating buildup. However, by maintaining a low-electrolyte suspension, hydrogen evolution was minimized.

Another difficulty occurred which was associated with the nonrigid attachment of the grid to the mounting ring. On removal of the assembly from the furnace after its heat treatment at 537°C for 10 minutes (to develop an oxide film on the molybdenum) the grid warped or moved on cooling, causing the glass coating to "pop off" where the grid and ring overlapped.

The large grids were coated in general by the same techniques as used for coating the 7-cm grids. While several voltage-current conditions could be used, 200 volts and 5.5 amperes were applied for 15 seconds in coating the two grids supplied. The separation of the anode and cathode was 11.4 cm. The grids were fired in a vertical position in accordance with the usual procedures. The fired grids contained, in one case, two filled holes and, in the other, 12 filled holes. Their general appearance was comparable to the 7-cm grids coated in Phase II. The fused glass coating thickness was typically 0.048 cm.

DISCUSSION OF RESULTS

Proper control of all of the parameters involved in the electrodeposition process made it feasible to apply glass coatings to metal substrates having the configuration of the grids under study. Optimum deposition conditions involved not only the electrophoretic process wherein charged solid particles of glass were the only current carriers. A small but significant degree of ionic conduction enhanced the total coating process through a bonding mechanism which caused the deposited glass particles to adhere to the molybdenum substrate and to each other.

The overall electrochemical processes involved were not subject to study in the program described, but observation during and after the process indicated the required conditions for successful coating application. Under proper deposition conditions as described by the voltage-current parameters, glass particles would migrate to the molybdenum anode electrophoretically. Simultaneously, molybdenum atoms would become ionized and hydrogen gas would be evolved at the cathode in small amounts. The darkened molybdenum surface and a slight bluish color of the deposited glass coating indicated that the positively charged molybdenum ions, in part, combined with oxygen ions (to form an oxide coating on the substrate), and in part migrated into the glass-powder coating to flocculate the particles and bond them together.

Though the electrochemical mechanisms proposed were not confirmed, it was observed that when all other parameters such as deposition time, specific gravity of the

glass-water suspension, etc. were maintained, the voltage-current parameters could be used to define the optimum coating conditions.

A second parameter requiring attention is that of the characteristics of the glass-water suspension. Where it would be desirable to coat a relatively large number of grids, a relatively large deposition tank should be used in order to have a stable glass-water suspension. Each time a grid is coated it removes glass from the suspension to change the glass-water ratio and to contribute ions to the water. These changes are not so significant when but a few grids are to be coated, but in a production process, corrections would have to be made to maintain proper suspension characteristics. Glass additions would be required periodically as would replacement of some of the ionized water by distilled water. These adjustments are particularly important in view of the rather rigid thickness requirements of the glass coating.

CONCLUSIONS

The application of a glass coating to molybdenum ion accelerator grids can be accomplished by the electrodeposition process. Furthermore, the total 0.056-cm-thick coating required could be applied in a single deposition process.

The parameters that must be controlled for successful deposition include:

(1) Specific gravity of water-glass suspension

(2) Applied d-c voltage

(3) Current

(4) Deposition time

(5) Cathode-anode spacing.

A feature of the coating process which was beneficial to achieving the required coating thickness was that of depositing the coating on the hole walls and both faces of the grid. Though the coating on the back face of the grid was removed before the fusing process, its presence during deposition enabled a maximum buildup of coating on the hole wall surfaces.

The electrodeposited coatings could be fused to the molybdenum grid by the same atmosphere-heat treatment used to give bubble-free coatings when application was by the wet-spraying process.

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